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IS 13105 (1991): 2-Amino-5-[N-Ethyl-N-(2-Methane Sulphonamido Ethyl)] Aminotoluene, Sesqui Sulphate (Monohydrate), Photographic Grade (CD-3) [CHD 5: Electroplating Chemicals and Photographic Materials]

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भारतीय मानक

2-एमिनो-5-[एन-इथाइल-एन-(2-मीथेन सल्फोनामाइडो इथाइल)] एमिनोटॉलूईन, सेस्क्वी सल्फेट (मोनोहाइड्रेट), फोटोग्राफिक ग्रेड (सीडी-3)

Indian Standard

2-AMINO-5-[N-ETHYL-N-(2-METHANE SULPHONAMIDO ETHYL)] AMINOTOLUENE, SESQUI SULPHATE (MONOHYDRATE), PHOTOGRAPHIC GRADE (CD-3)

UDC 547·553·2 : 771·7

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

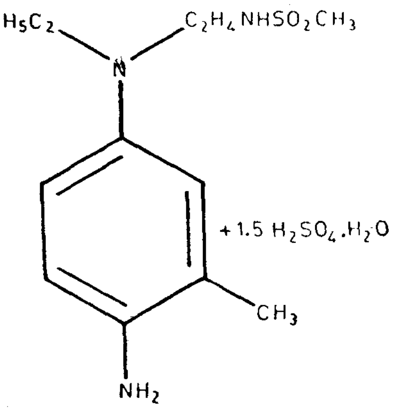
July 1991 **Price Group 3**

Photographic Materials Sectional Committee, CHD 04

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

2-Amino-5-[N-ethyl-N-(2-methane sulphonamido ethyl)] aminotoluene sesqui sulphate (monohydrate), (CD-3), photographic grade is used as a photographic colour developer. It is also known in the trade by names like colour developer-3 or CD-3. It has the following structural formula:



(Molecular mass = 436·52)

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

2-AMINO-5-[N-ETHYL-N-(2-METHANE SULPHONAMIDO ETHYL)] AMINOTOLUENE, SESQUI SULPHATE (MONOHYDRATE), PHOTOGRAPHIC GRADE (CD-3)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for 2-amino-5-[N-ethyl-N-(2-methane sulphonamido ethyl)] aminotoluene, sesqui sulphate (monohydrate), photographic grade (CD-3), used as the basic constituent of developing solutions for treatment of multilayer light sensitive materials of colour photography and cinematography.

2 REFERENCES

The Indian Standards listed below are the necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Water for general laboratory use (*second revision*)
2263 : 1979	Methods of preparation of indicator solution for volumetric analysis (*first revision*)
4905 : 1968	Methods for random sampling
6128 : 1971	Desiccators

3 REQUIREMENTS

3.1 Description

The material shall be in the form of light pinkish brown crystalline powder, free from agregates or large crystals and shall consist essentially of 2-amino-5-[N-ethyl-N-(2-methane sulphonamido ethyl)] aminotoluene, sesqui sulphate (monohydrate), photographic grade (CD-3).

3.2 Solution in Water

A freshly prepared solution of the material containing 5 g of the sample in 100 ml of water shall be clear and free from sediments.

3.3 The material shall also comply with the requirements prescribed in Table 1 when tested according to the methods given in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the table.

4 PACKING, MARKING AND SAFETY PRECAUTIONS

4.1 Packing

The material shall be packed in suitable, dark coloured airtight bottles or as agreed to between the purchaser and the supplier.

Table 1 Requirements for 2-Amino-5-[N-Ethyl-N-(2-Methane Sulphonamido Ethyl)] Aminotoluene, Sesqui Sulphate (Monohydrate), Photographic Grade (CD-3)

(*Clause* 3.3)

Sl No.	Characteristics	Requirements	Method of Test (Ref to Cl No. in Annex A)
(1)	(2)	(3)	(4)
i)	Free base, percent by mass	62·0 ± 0·5	A-2
ii)	Sulphuric acid, percent by mass	33·5 ± 0·5	A-3
iii)	Ash, percent by mass, *Max*	0·1	A-4
iv)	Iron (as Fe), percent by mass, *Max*	0·001	A-5
v)	Heavy metals (as Pb), percent by mass, *Max*	0·001	A-6
vi)	Volatile matter at 70°C, percent by mass, *Max*	0·2	A-7
vii)	*p*H of 5 percent aqueous solution	1·5 ± 0·2	A-8
viii)	Identity test	To pass melting point test	A-9

4.2 Marking

The containers shall be securely closed and marked legibly and indelibly with the following information:

a) Name and grade (photographic grade) of the material,
b) Mass of the material,
c) Date of manufacture,
d) Indication of the source of manufacture, and
e) Lot number in code or otherwise.

4.3 Safety Precautions

2-Amino-5-[N-ethyl-N-(2-methane sulphonamido ethyl)] aminotoluene, sesqui sulphate (monohydrate), photographic grade (CD-3), has a pronounced general toxic action and shows particular effect on liver and central nervous system. On skin contract, it may cause eczema.

4.3.1 Work with this material should be done only with protective clothing. The place should be provided with exhaust and ventilation systems.

5 SAMPLING

Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in Annex B.

ANNEX A

(*Clause* 3.3 and *Table* 1)

METHODS OF TEST FOR 2-AMINO-5-[N-ETHYL -N-(2-METHANE SULPHONAMIDO ETHYL)] AMINOTOLUENE, SESQUI SULPHATE (MONOHYDRATE), PHOTOGRAPHIC GRADE (CD-3)

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the result of analysis.

A-2 DETERMINATION OF FREE BASE

A-2.0 General

Using standard ceric sulphate solution, the free base content is found out by volumetric method.

A-2.1 Reagents

A-2.1.1 *Dilute Sulphuric Acid* — 1 N.

A-2.1.2 *Dilute Hydrochloric Acid* — 3 N.

A-2.1.3 *Ferroin Indicator* — *See* IS 2263 : 1979.

A-2.1.4 *Standard Ceric Sulphate Solution* — 0·05 N.

A-2.2 Procedure

Weigh accurately about 1·25 g of sample in a 250 ml volumetric flask. Dissolve it in dilute sulphuric acid and make up to the mark with dilute sulphuric acid.

A-2.2.1 In a 1 000 ml conical flask, take 100 ml of dilute hydrochloric acid 200 ml of water and add 10 drops of ferroin indicator. Titrate with ceric sulphate solution to a pale blue end point. Record the blank titre value V_1. Pipette out 50 ml of the sample solution prepared in **A-2.2**, into the conical flask and continue titration without refilling the burette until a loss of orange colour is observed momentarily. Reduce the rate of addition as end point is approached so that one drop is added every 3 to 4 seconds near the end point. The end point is of a greenish yellow colour which does not change with the addition of one more drop. Record the net titre value V.

A-2.3 Calculation

$$\text{Free base, percent by mass} = \frac{(V - V_1) \times F \times 3{\cdot}392\,5}{M}$$

where

V = net titre value in ml,

V_1 = blank titre value in ml,

F = factor of 0·05 N ceric sulphate solution, and

M = mass in g of the sample taken for the test.

A-3 DETERMINATION OF SULPHURIC ACID

A-3.0 General

Sulphates are precipitated using barium chloride and determined gravimetrically. From this, the sulphuric acid content is found out.

A-3.1 Reagents

A-3.1.1 *Barium Chloride Solution* — approximately 10 percent (*m/v*).

A-3.1.2 *Concentrated Hydrochloric Acid*

A-3.2 Procedure

Weigh accurately 5 ± 0·1 g of the sample, transfer it to 100 ml volumetric flask, dissolve and make up to the mark using water. Pipette out 20 ml of this solution into a 250 ml of beaker containing 80 ml of water and one millilitre of hydrochloric acid and heat to boil. To this hot solution, add about 10 ml of hot barium chloride solution and continue stirring at boiling temperature for 2 minutes to complete the precipitation of barium sulphate. Continue heating on a water bath for 1-2 hours and filter through Whatman No. 40 filter paper. Wash the precipitate by hot water until the filtrate is free from chloride. Incinerate and ignite the precipitate in a tared silica crucible. Cool the crucible to room temperature and weigh to a constant mass.

A-3.3 Calculation

$$\text{Sulphuric acid, percent by mass} = M \times 0{\cdot}42 \times \frac{100}{20} \times \frac{100}{M_1}$$

where

M = mass in g of the precipitate, and

M_1 = mass in g of the material taken for the test.

A-4 DETRMINATION OF ASH

A-4.1 Apparatus

A-4.1.1 *Silica Crucible* — Provided with lid.

A-4.1.2 *Furnace* — Preferably with automatic temperature control device.

A-4.1.3 *Desiccator* — *See* IS 6128 : 1971.

A-4.2 Procedure

Weigh accurately 5·000 g of the material and transfer to tared silica crucible and then ignite at dull red heat (approximately 600°C). Cool in a desiccator and weigh to a constant mass. Preserve the residue for the determination of iron and heavy metals in **A-5** and **A-6** respectively.

A-4.3 Calculation

$$\text{Ash, percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the material taken for the test.

A-5 DETERMINATION OF IRON

A-5.0 General

Two methods have been prescribed for the determination of iron, namely (a) *o*-phenanthroline method, and (b) thioglycollic acid method. However, in case of dispute, the *o*-phenanthroline method shall be the referee method.

A-5.1 *o*-Phenanthroline Method

A-5.1.0 *General*

o-phenanthroline, hydroxylamine hydrochloride solution and acetate buffer solution of *p*H 5 in equal quantities are mixed with the material and the colour is compared with that produced by standard iron solution under identical conditions.

A-5.1.1 *Reagents*

A-5.1.1.1 *Acetate buffer* — *p*H 5.

Add 23 g of anhydrous sodium acetate to 58 ml of 2 molar acetic acid and dilute to 1 000 ml with water. Adjust the final *p*H of the solution to 5 ± 0·1 with acetic acid or 10 percent sodium hydroxide solution.

A-5.1.1.2 *Hydroxylamine hydrochloride* — 10 percent aqueous solution (*m*/*v*).

A-5.1.1.3 *o-phenanthroline (1 : 10 o-phenanthroline)* — 0·1 percent solution (*m*/*v*).

A-5.1.1.4 *Standard iron solution*

Dissolve 7·02 g of crystalline ferrous ammoium sulphate [$FeSO_4 . (NH_4)_2 (SO_4)_2 . 6H_2O$] in about 300 ml of water containing 2 ml of concentrated sulphuric acid and dilute to 1 000 ml with water. Further dilute 5 ml of this solution to 1 000 ml. One millilitre of the diluted solution contains 0·005 mg of iron (as Fe). This dilution shall be done afresh when required.

A-5.1.2 *Procedure*

A-5.1.2.1 Add 5 ml of concentrated hydrochloric acid to the residue obtained from the determination of ash (*see* **A-4**) and evaporate to dryness. Dissolve the residue in 15 ml of dilute hydrochloric acid and warm on a steam-bath for 10 minutes. Cool, dilute to 50 ml with water in a volumetric flask and mix thoroughly. Use 20 ml of this solution for the determination of iron and preserve the remaining solution for heavy metals test in **A-6**.

A-5.1.2.2 Thoroughly mix equal parts of *o*-phenanthroline, hydroxylamine hydrochloride and acetate buffer of *p*H 5. Add 5 ml of this mixture to each of 4 ml of the standard iron solution and 20 ml of sample solution. Dilute to 50 ml with water and mix well.

A-5.1.2.3 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced in the sample solution is not deeper than that produced in the standard iron solution.

A-5.2 Thioglycollic Acid Method

A-5.2.0 *General*

Concentrated hydrochloric acid is added to the residue obtained from the determination of ash, evaporate to dryness and finally dissolved in dilute hydrochloric acid. Thioglycollic acid is then added to the solution, the solution is made alkaline and the colour produced is compared with that produced by standard iron solution under identical conditions.

A-5.2.1 *Apparatus*

A-5.2.1.1 *Nessler cylinders* — 50 ml capacity.

A-5.2.2 *Reagents*

A-5.2.2.1 *Dilute hydrochloric acid* — 10 percent (*v*/*v*).

A-5.2.2.2 *Thioglycollic acid* — purity, 89 percent, *Min*.

A-5.2.2.3 *Dilute ammonium hydroxide* — 1 : 2.

A-5.2.2.4 *Citric acid solution* — 25 percent (*m*/*v*).

A-5.2.3 *Procedure*

A-5.2.3.1 Transfer 20 ml aliquot of the sample solution from **A-5.1.2.1** to a Nessler cylinder and dilute to about 30 ml. Into the second Nessler cylinder containing 5 ml of dilute hydrochloric acid pipette out 4 ml of standard iron solution and dilute to about 30 ml with water. To both the cylinders, add 2 ml of citric acid solution and 2 to 3 drops of thioglycollic acid. Mix, make just alkaline with ammonium hydroxide and dilute to 50 ml.

A-5.2.3.2 The material shall be taken to have not exceeded the limit prescribed for iron, if the colour produced in the sample solution is not deeper than that produced in the standard iron solution.

A-6 DETERMINATION OF HEAVY METALS

A-6.0 General

The colour produced by heavy metals in the sample on treating with hydrogen sulphide is compared with that produced with standard lead solution under identical conditions.

A-6.1 Reagents

A-6.1.1 *Dilute Ammonium Hydroxide* — 10 percent (*m/m*).

A-6.1.2 *Dilute Hydrochloric Acid* — approximately 0·1 *N*.

A-6.1.3 *D-Nitrophenol Indicator* — 0·25 percent aqueous solution (*m/v*).

A-6.1.4 *Hydrogen Sulphide* — saturated aqueous solution.

A-6.1.5 *Standard Lead Solution*

Dissolve 0·64 g of lead nitrate in water, add one millilitre of concentrated nitric acid and dilute to 1 000 ml with water. Further dilute 10 ml of this solution to 1 000 ml. One millilitre of the resulting solution contains 0·004 mg of Pb.

A-6.2 Procedure

A-6.2.1 To the 20 ml portion of the sample solution (*see* **A-5.1.2.1**) in a Nessler cylinder, add a few drops of *p*-nitrophenol indicator and then add ammonium hydroxide dropwise, until the solution turns yellow. Add dilute hydrochloric acid dropwise, until the solution becomes colourless again and then add 0·5 ml in excess. Add 5 ml of hydrogen sulphide solution and dilute to 50 ml.

A-6.2.2 At the same time transfer 5 ml of standard lead solution into another Nessler cylinder and make it neutral to *p*-nitrophenol indicator by using dilute ammonium hydroxide and dilute hydrochloric acid as described in **A-6.2.1**. Add 0·5 ml of dilute hydrochloric acid in excess and 5 ml of hydrogen sulphide solution. Dilute to 50 ml.

A-6.2.3 The material shall be considered to have passed the requirement of the test if the colour produced in the sample solution is not deeper than that produced in the standards solution.

A-7 DETERMINATION OF VOLATILE MATTER

A-7.0 General

A known quantity of the material is dried at 70°C and loss in mass is taken as volatile matter.

A-7.1 Apparatus

A-7.1.1 *Glass-Stoppered Shallow Weighing Bottle*

A-7.1.2 *Oven* — preferably with automatic temperature control device.

A-7.1.3 *Desiccator* — *See* IS 6128 : 1971.

A-7.2 Procedure

Weigh accurately 3 ± 0·1 g of the material in the weighing bottle and dry in the oven at 70°C for about 8 hours. Cool in the desiccator and weigh.

A-7.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{(M - M_1) \times 100}{M}$$

where

M = mass in g of the material taken for the test, and

M_1 = mass in g of the material obtained on drying.

A-8 DETERMINATION OF *p*H

A-8.0 General

The *p*H of a 5 percent solution of the material is determined by using a suitable *p*H meter.

A-8.1 Apparatus

A-8.1.1 *pH Meter* — with glass and calomel electrodes.

A-8.2 Procedure

Prepare a five percent solution of the material in a 100 ml beaker, using freshly boiled and cooled water. Measure the *p*H of the solution using a *p*H meter.

A-9 IDENTITY TEST

A-9.1 Melting Point Test

Assemble capillary tube melting point apparatus using a thermometer that covers the range 100-200°C. Prepare 3 capillary tubes, placing in them (1) the sample to be tested, (2) a sample known to be 2-amino-5-N [N-ethyl-N-(2-methane sulphonamido ethyl)] aminotoluene, sesqui sulphate (monohydrate), photographic grade (CD-3), and (3) a finely ground mixture of (1) and (2) mixed in the ratio of 1 : 1. Identify the tubes, attach them to the thermometer and insert in the melting point bath. Heat the bath to approximately 115°C and thereafter heat at a constant rate of 1°C per minute stirring constantly. Note the melting point of the contents of each tube as indicated by the first appearance of liquefaction. The melting point of the mixture shall be not lower than those of the two samples. The melting point of the sample should be between 123°C and 130°C.

ANNEX B

(*Clause* 5)

SAMPLING OF 2-AMINO-5-[N-ETHYL-N-(2-METHANE SULPHONAMIDO ETHYL)] AMINOTOLUENE, SESQUI SULPHATE (MONOHYDRATE), PHOTOGRAPHIC GRADE (CD-3)

B-1 GENERAL REQUIREMENTS FOR SAMPLING

B-1.1 In drawing, preparing, storing and handling test samples, the following precautions shall be observed.

B-1.2 Samples shall not be taken out at a place exposed to weather.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled the sampling instrument and containers for samples from adventitious contamination.

B-1.4 To draw representative samples, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and airtight glass or plastic containers.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and the year of manufacture.

B-1.8 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

B-2 SCALE OF SAMPLING

B-2.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lot.

B-2.2 From each lot five containers shall be selected (*see* **B-2.2.1**) for drawing samples for test. In case the lot contains less than five containers, each container shall be tested.

B-2.2.1 These containers shall be selected at random from the lot. In order to ensure the randomness of selection, procedure given in IS 4905 : 1968 may be followed. In case this standard is not available, the following procedure may be adopted:

> Arrange all the containers in the lot in a systematic manner and, starting from any one, count them as 1, 2,.........r, where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample.

B-3 PREPARATION OF TEST SAMPLES

B-3.1 Drawing of Samples

Draw a representative sample from each container selected.

B-3.1.1 Mix portions of the material drawn from the same container to prepare the test sample representing the container. The quantity of material to be drawn from each container shall be sufficient to conduct all the tests given in **3** and shall be not less than 200 g.

B-3.1.2 The samples shall be stored in dry glass bottles or other suitable containers, sealed airtight and labelled with all the particulars of sampling given in **B-1.7**.

B-3.2 The remaining portions of the material from each of the five containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the container samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 All the individual and composite samples shall be immediately transferred to separate sample containers and labelled with full identification particulars.

B-3.4 The referee samples consisting of a composite sample and a set of individual samples, shall bear the seal of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

B-4 NUMBER OF TESTS

Tests for the determination of free base, heavy metals and iron shall be conducted on each of the five individual samples. Tests for all other characteristics prescribed in **3** and Table 1 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 Tests Carried Out on Five Individual Test Samples

B-5.1.1 *Free Base*

From the test results for free base, the mean ($\bar{X}$) and the range (R) of test results shall be calculated (range being defined as the difference between the maximum and the minimum values of test results). The lot shall be considered to have passed in respect of assay, if the value of the expression ($\bar{X} \pm 0.6\,R$) lies between 61·5 and 62·5.

B-5.1.2 *Heavy Metal and Iron*

The lot shall be considered to have passed for the characteristics tested if each test sample passes the corresponding test prescribed.

B-5.2 Test Carried Out on Composite Test Sample

The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample (*see* **B-4.1**) if the composite test sample satisfies the corresponding test prescribed.

B-5.3 The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **B-5.1** and **B-5.2**.

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